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Indian Standard

METHODS OF TEST FOR VITREOUS ENAMELWARE

PART 2 TEST METHODS

Section 1 Resistance to Citric Acid at Room Temperature and Boiling Temperature

(First Revision)

UDC 666·293 : 620·193·41 : 661·734·1

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Indian Standard

METHODS OF TEST FOR VITREOUS ENAMELWARE

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(First Revision)

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(*Continued on page* 14)

Indian Standard

METHODS OF TEST FOR VITREOUS ENAMELWARE

PART 2 TEST METHODS

Section 1 Resistance to Citric Acid at Room Temperature and Boiling Temperature

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 30 November 1985, after the draft finalized by the Ceramicware Sectional Committee had been approved by the Chemical Division Council.

0.2 The Committee, while reviewing IS:3972-1968*, decided to publish it in two parts. Part 1 deals with production of specimens for testing in two sections, namely, Section 1 Enamelled sheet steel, and Section 2 Enamelled cast iron. Part 2 deals with various test methods applicable to vitreous enamelled sheet steel and vitreous enamelled cast iron. The Committee also decided that Part 2 shall have various sections and each section would deal with a particular test method.

0.3 In the preparation of this standard (Part 2/Sec 1) assistance has been derived from the following standards issued by the International Organization for Standardization (ISO):

ISO 2722-1973	Vitreous and porcelain enamels — Determination of resistance to citric acid at room temperature.
ISO 2742-1973	Vitreous and porcelain enamels — Determination of resistance to boiling citric acid.
ISO 2733-1983	Vitreous and porcelain enamels — Apparatus for testing with acid and neutral liquids and their vapours.

---

*Methods of test for vitreous enamelware.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS:2-1960*.

1. SCOPE

1.1 This standard prescribes the methods of test for determining the resistance to citric acid at room temperature and boilng temperature applicable to vitreous enamelled sheet steel and vitreous enamelled cast iron wares.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS:2717-1979† shall apply.

3. SAMPLING

3.1 Representative samples of vitreous enamelware shall be drawn as specified in individual material specifications.

4. QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS: 1070-1977‡) shall be used where the use of water as a reagent is intended.

> NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis or test.

5. TEST FOR RESISTANCE TO CITRIC ACID AT ROOM TEMPERATURE

5.0 General — This test method is not suitable for matt enamels, enamels that come in contact with hot and strong acids and enamelled articles that are used in the chemical industry.

5.0.1 *Outline of Method* — A part of the specimen is exposed to attack by a 10 percent (*m/m*) solution of citric acid for 15 minutes ± 30 seconds. The results are based on the appearance and cleanability of the enamelled surface.

5.1 Apparatus

5.1.1 *Graduated Measuring Cylinder, Capacity 100 ml* — *See* IS : 878-1975§.

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*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to vitreous enamelware and ceramic-metal systems (*first revision*).

‡Specification for water for general laboratory use (*second revision*).

§Specification for graduated measuring cylinders (*first revision*).

5.1.2 *Pipette — See* IS : 1117-1975*.

5.1.3 *Filter Paper*

5.1.4 *Towel, White* — Cotton or flax.

5.1.5 *Pencil* — Conventional graphite HB pencils hardness conforming to IS : 1375-1959†.

5.1.6 *Watch-Glass* — made of polyethylene or glass external diameter approximately 30 mm.

5.1.7 *Balance* — accurate to ±0·1 g.

5.1.8 *Electric Lamp* — 40 W, inside white (for example, siliconized).

5.2 Reagents

5.2.1 *Citric Acid Solution* — Dissolve 10 g pure crystalline citric acid ($C_6H_8O_7.H_2O$) in 100 ml of distilled water. A fresh solution, prepared the same day, is required for each test.

5.2.2 *Grease Solvent* — Acetone or trichloroethylene, suitable for cleaning the specimen.

5.3 Preparation of Test Specimen — Each specimen shall be prepared in accordance with IS : 3972 (Part 1/Sec 1)-1982‡ or IS : 3972 (Part 1/Sec 2)-1982§ depending upon the base metal used for enamelling. The specimen shall be cleaned with the grease solvent (**5.2.2**), rinsed with hot water until the water spreads evenly on the surface and then dried with the clean towel (**5.1.4**); it shall be dabbed, not rubbed.

5.4 Procedure

5.4.1 *Attack by the Test Solution* — Place a filter paper of a small diameter (10-15 mm) on the test specimen and then place a few drops of the test solution (**5.2.1**) on the filter paper to keep it in moistened condition and cover the etching zone with watch glass. Care should be taken so that the wet filter paper does not dry out. After 15 minutes ± 30 seconds remove the watch glass, wash the specimen with either distilled or running water, then dry it with filter paper without piping. Care shall be taken that when using running water, no film of water is allowed to form.

---

*Specification for one mark pipettes (*first revision*).

†Specification for black lead pencils (*first revision*).

‡Methods of test for vitreous enamelware: Part 1 Production of specimens for testing, Section 1 Enamelled sheet steel (*first revision*).

§Methods of test for vitreous enamelware: Part 1 Production of specimens for testing, Section 2 Enamelled cast iron (*fisst revision*).

5.4.2 *Determination* — Each test specimen shall be examined within 2 hours after the exposure to the test solution. For evaluation, only that part of the surface of the specimen which has been subjected to attack by acid shall be considered as a treated area.

The evaluation is based on the examinations which shall be in accordance with the scheme and results given below. The specimen is to be assigned to one of the five classes designated as AA, A, B, C and D.

5.4.2.1 *Visual examination* — View the different areas at varying angles, at a distance of 250 mm from the specimen without a magnifying glass, in order to ascertain whether or not the treated area differs from the non-treated area (for instance, if the brightness or the colour has changed). Carry out the examination in daylight, avoiding direct sunlight. The specimen may also be examined in artificial light provided the latter is uniform and strong enough. If the treated area differs in any respect from the non-treated area, the specimen fails the test.

5.4.2.2 *Pencil test (dry)* — Draw some nearly parallel lines on both the treated and non-treated areas with a pencil (**5.1.5**). If, on rubbing the specimen with the dry towel (**5.1.4**), the lines on the treated area are more difficult to remove than those on the non-treated area, the specimen fails the test.

5.4.2.3 *Reflection test* — Set the specimen in such a way that the image of the test lamp (**5.1.8**) 35 ± 10 cm away from the specimen reflects on the non-treated area with an angle of incidence of 45°. Then watch the image of the lamp on the non-treated area while the specimen is slowly removed, so that the image of the lamp moves into the treated area. If no blurring of the image is observed while it passes from one area to the other, the specimen passes the test. If such is not the case, it is necessary to distinguish, in the passage from one area to the other, if there is a blurring or a complete disappearance of the image.

5.4.2.4 *Pencil (test moist)* — Repeat the test specified in **5.4.2.2** using the towel (**5.1.4**) which has been moistened with water and thoroughly wrung out, without using any soap or detergent. If the lines on the treated area are more difficult to remove than those on the non-treated area, the specimen fails the test.

5.5 Classification of Results

5.5.1 In consequence of the determinations which have been performed in accordance with **5.4.2** the enamels are divided into five classes which are shown as class AA, A, B, C and D, in Fig. 1 and Table 1.

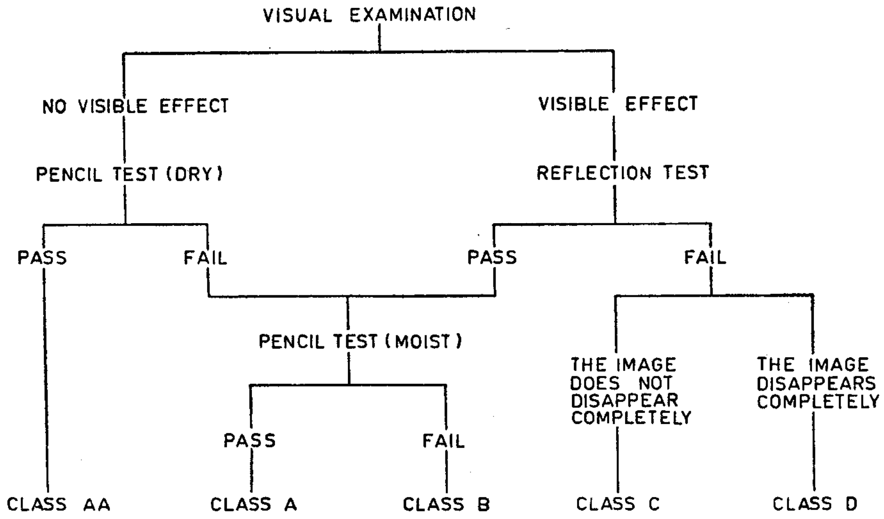


FIG. 1 FLOW SHEET OF TEST PROCEDURE FOR CLASSIFICATION OF TREATED SPECIMENS

TABLE 1 CRITERIA FOR RESPECTIVE CLASSES OF RESISTANCE TO CITRIC ACID AT ROOM TEMPERATURE

(*Clause* 5.5.1)

CLASS	VISUAL EXAMINATION	REFLECTION TEST	TEST WITH PENCIL	
			Dry	Moist
(1)	(2)	(3)	(4)	(5)
AA	PASS	—	PASS	—
A	PASS	—	FAIL	PASS
	FAIL	PASS	—	PASS
B	PASS	—	FAIL	FAIL
	FAIL	PASS	—	FAIL
C	FAIL	PARTLY FAIL	—	—
D	FAIL	TOTALLY FAIL	—	—

5.6 Test Report

5.6.1 The test report shall include the following particulars:

a) Description of the specimen,

b) Results of the tests specified in **5.4**, and

c) Classification of the enamel coating according to **5.5**.

6. TEST FOR RESISTANCE TO BOILING CITRIC ACID

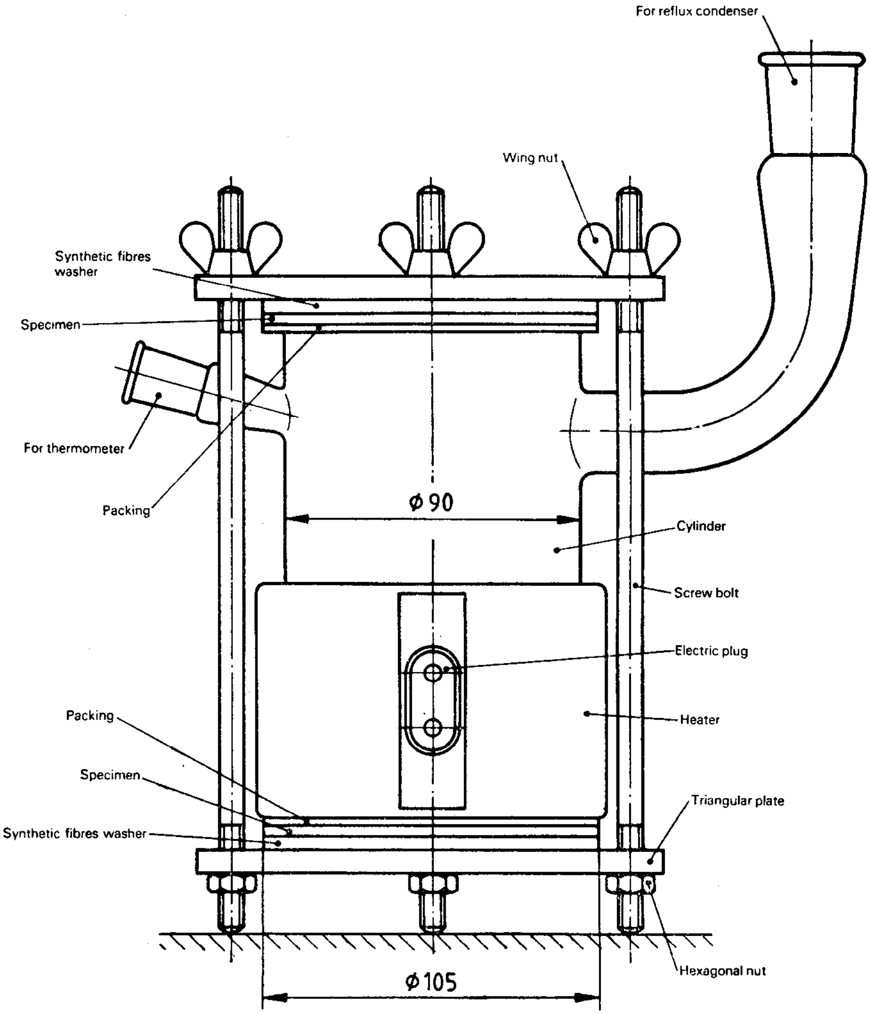
6.1 General — This test is especially intended for the testing of enamels for articles which are used for boiling, slightly dissociated acids, for example, cooking utensils but not suitable for enamels used in the chemical industry or those exposed to heavy attack by inorganic acids for a long period.

NOTE — This method of test is also suitable for determining the chemical resistance of enamels to other acid solutions and permits also, if desired, the determination of the resistance of enamels to the vapour phase of the respective test solution.

6.1.1 *Outline of the Method* — The enamelled specimen is exposed to attack by a boiling 6 percent (*m/m*) solution of citric acid for 2·5 hours. The loss in mass is determined and therefrom the loss in mass per unit area is calculated. The lower the loss in mass per unit area, the higher is the resistance of the vitreous enamel to boiling citric acid.

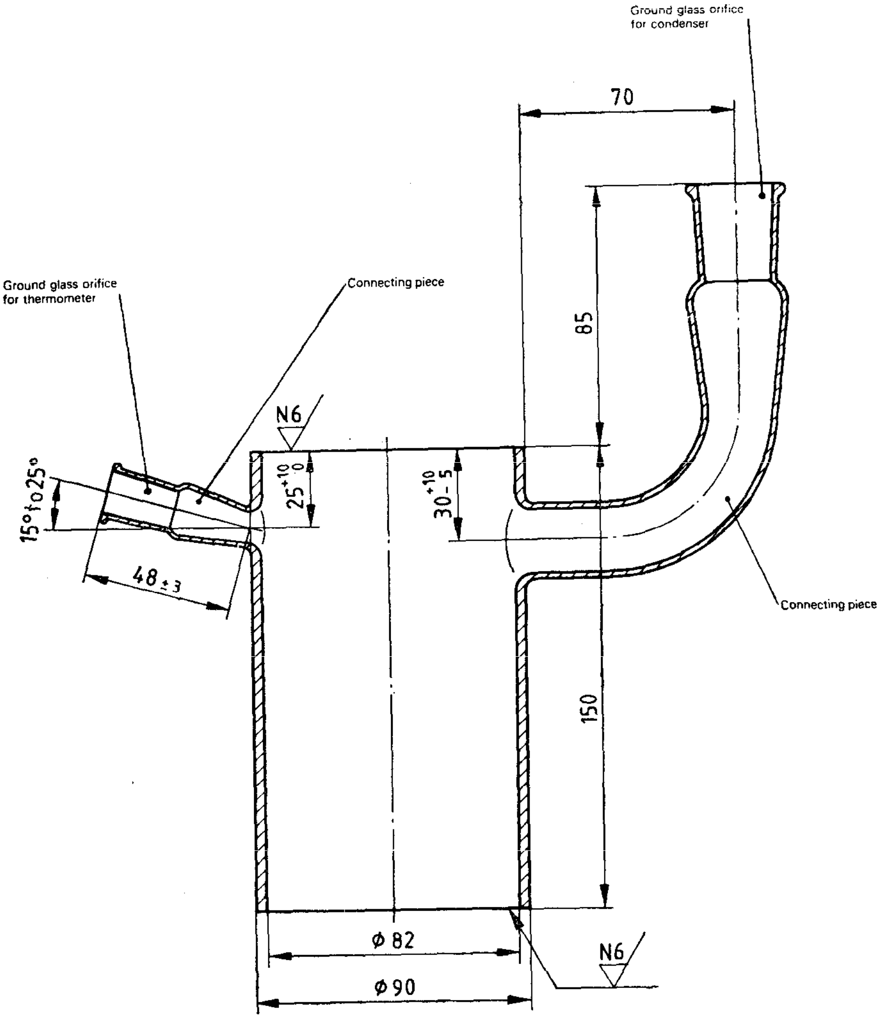
NOTE — If information is desired on the further corrosion, the attack by a boiling solution of citric acid may be extended to 6, 24 more hours.

6.1.2 *Testing Apparatus* — The testing apparatus shall be similar to one given in Fig. 2. It consists of a cylinder as shown in Fig. 3, made from borosilicate glass, with two adjacent supports with standard sockets for holding on one side a reflux condenser (with a nominal length of 400 mm and standard ground joint) and on the other side a thermometer (having a range of 0 to 250°C and with a graduation interval of 1°C) for measuring the temperature of the vapour chamber. The cylinder which holds the test specimens is supported between two triangular plates (*see* Fig. 4) locked at the corners by the screw bolts, three wing nuts and three hexagonal nuts (all made from a corrosion resistant steel). Synthetic fibres washers (resistant to citric acid and water at 140°C) are provided for fixing between the triangular plates and specimen. Packing rings (100 mm external diameter, $80^{+0}_{-0\cdot3}$ mm internal diameter, 2 to 3 mm thick, made from rubber such as chloroprene with hardness 70 IRHD, resistant to citric acid and water at 140°C, are provided for realising the specimen against the ground edges of the cylinder or protective envelope as shown in Fig. 5. The lower half of the apparatus is provided with a 500 W heater and heat control device. The dimensions of the heater are such that the upper rim of the heater is 95 mm from the lower edge of the cylinder and the heater does not touch the packing.



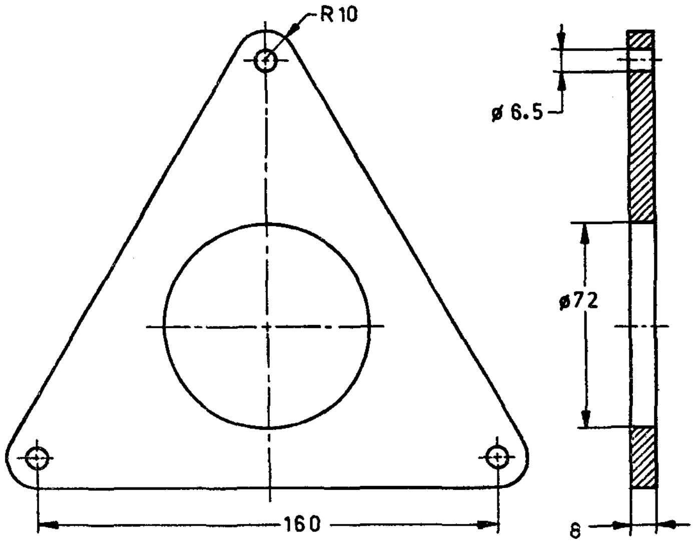
All dimensions in millimetres.

FIG. 2 TESTING APPARATUS



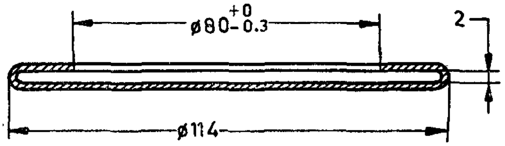
All dimensions in millimetres.

FIG. 3 CYLINDER



All dimensions in millimetres.

FIG. 4 TRIANGULAR PLATE



All dimensions in millimetres.

FIG. 5 PROTECTIVE ENVELOPE

6.1.3 *Hot Air Oven* — capable of maintaining a temperature of at least 130°C.

6.1.4 *Desiccator* — with an internal diameter of 200 mm.

6.1.5 *Graduated Measuring Cylinder*—500 ml capacity (*see* IS : 878-1975*).

6.1.6 *Beakers* — *See* IS : 2619-1971†.

6.1.7 *Balance* — accurate to ±0·2 mg.

6.1.8 *Sponge* — soft.

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*Specification for graduated measuring cylinders (*first revision*).

†Specification for glass beakers.

6.2 Reagents

6.2.1 *Citric Acid, 6 Percent (m/m) Solution* — Dissolve 32 g of pure crystalline citric acid ($C_6H_8O_7.H_2O$) in 500 ml of distilled water. A fresh solution prepared the same day is required for each test.

6.2.2 *Grease Solvent* — Acetone or trichloroethylene suitable for cleaning the specimen.

6.3 Preparation of Test Specimen — Each specimen of approximately 105 mm diameter shall be prepared in accordance with IS:3972 (Part 1/ Sec 1)-1982* or IS:3972 (Part 1/Sec 2)-1982† depending upon the base metal used for enamelling. Clean the specimen with the grease solvent (**6.2.2**), rinse with hot water until the water spreads evenly on the surface. The specimen shall be dried for 2 hours in the hot-air oven at 110 ± 5°C, then cooled in the desiccator and weighed to the nearest 0·2 mg.

6.3.1 For each determination two tests shall be carried out.

6.4 Procedure

6.4.1 Fix the specimen in the testing apparatus (**6.1.1**) so that it forms the bottom of the cylinder in such a way that the enamel coated side of the specimen is facing the interior of the cylinder. The top of the cylinder is formed by another specimen with the same enamel coating or by a stainless steel plate approximately 105 mm diameter. Screw down the three wing nuts evenly to make the testing apparatus watertight.

6.4.2 Add 350 ml of the test solution (**6.2.1**) into the cylinder through socket, replace the reflux condenser and switch on the heater. As soon as the test solution begins to boil (two to four bubbles per second) lower the current by the rheostat control so that the test solution simmers during the remainder of the test. Record the temperature during the simmering.

6.4.3 The simmering time shall be 2·5 hours. If information is desired on the further corrosion, the simmering time may be extended to 6—24 or more hours. The experience has shown that the attack diminishes considerably with time.

6.4.4 After simmering for 2·5 hours or longer, as appropriate, empty the cylinder, and after cooling, rinse with distilled water. Take the specimen from the testing apparatus and wipe it three times with the sponge (**6.2.2**) and water. After carefully removing any packing residues from the edges of the specimen, dry it for 2 hours in the hot-air oven at 110 ± 5°C. After a further 2 hours in the desiccator, weigh it again to the nearest 0·2 mg (final mass).

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*Methods of test for vitreous enamelware: Part 1 Production of specimens for testing, Section 1 Enamelled sheet steel.

†Methods of test for vitreous enamelware: Part 1 Production of specimens for testing, Section 2 Enamelled cast iron.

6.5 Calculations

6.5.1 The loss of mass per unit area of the enamelled article after treatment with boiling citric acid is calculated as follows:

$$\text{Loss in mass, per square metre, } \triangle m = \frac{M_1 - M_2}{A}$$

where

M_1 = mass in g of the test specimen before test,

M_2 = mass in g of the test specimen after test, and

A = area in square metres of the exposed surface of the test specimen.

In order to distinguish between the testing results of different testing periods, the number of testing hours is to be stated as a subscript to the symbol, for example,

for 2·5 hours testing time, $\triangle m_{2\cdot5}$

for 6 hours testing time, $\triangle m_6$

for 24 hours testing time, $\triangle M_{24}$

6.5.2 The average of two results may be reported.

6.5.3 The results of the specimens which show defects such as pinholes, chipped edges or edge corrosion are omitted.

6.6 Test Report

6.6.1 The test report shall include the following particulars:

a) Testing temperature, in degrees Celsius;

b) Simmering time, in hours; and

c) Loss in mass per unit area.

(*Continued from page* 2)

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AMENDMENT NO. 1 JUNE 2007
TO
IS 3972 (PART 2/SEC 1) : 1985 METHODS OF TEST FOR VITREOUS ENAMELWARE

PART 2 TEST METHODS

Section 1 Resistance to Cirtic Acid at Room Temperature and Boilding Temperature

(First Revision)

(*Page* 4, *clause* **4.1**, *line* 2) — Substitute 'IS 1070 : 1992‡' *for* 'IS 1070 : 1977‡'.

(*Page* 4, *footnote marked* ‡) — Substitute the following for the existing:

'‡Reagent grade water — Specification (*third revision*).'

(*Page* 5, *clause* **5.1.5**, *line* 2) — Substitute 'IS 1375 : 1981†' *for* 'IS : 1375 - 1959†'.

(*Page* 5, *footnote marked* †) — Substitute the following for the existing:

'†Specification for black lead pencils (*first revision*).'

(*Page* 11, *clause* **6.1.6**, *line* 1) — Substitute 'IS 2619 : 1993†' *for* 'IS 2619 : 1971†'.

(*Page* 11, *footnote marked* †) — Substitute the following for the existing:

'†Glass beakers Specification (*second revision*).'

(CHD 9)

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